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Experiment 3 – Graphing Freezing Points

Discussion

The freezing and melting of a substance occur at the same temperature, which is measured when the solid and liquid phases are at equilibrium (therefore, the terms "melting point" and "freezing point" can be used interchangeably). Accurate measurement of this temperature is useful for identification purposes, since few chemicals share exactly the same melting point. The unofficial definition of freezing point is the temperature where crystals first start to form. Officially, freezing point is determined by graphing a "cooling curve", which is a plot of temperature versus time as a substance is being cooled. The temperature will decrease at first as the liquid is cooled; then the temperature will remain constant during the phase change from liquid to solid. This "plateau" is the freezing point. When all the liquid has frozen, the temperature will again decrease.

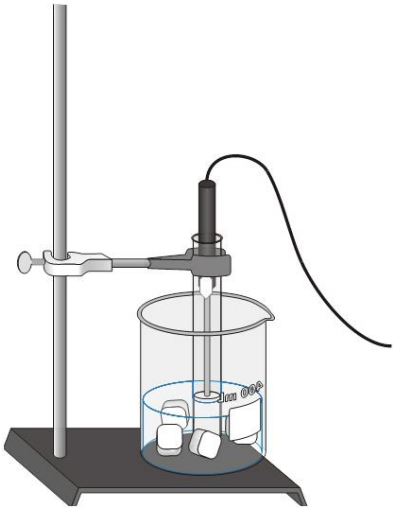
An impurity will disrupt the crystal structure of a substance; therefore, it must be cooled below its normal freezing point to achieve crystallization. This depression of the freezing point has some interesting applications: measuring the amount of impurity, identification by mixing a known with an unknown substance, lowering the freezing point of water using antifreeze, etc.

Occasionally, a substance will become colder than its normal freezing point and still remain a liquid. This unstable condition is called "supercooling". "Shocking" the system by stirring or jarring usually will cause crystallization and the substance will rapidly rise to its normal freezing point.

The goal of this experiment is to determine freezing points by graphing two cooling curves: (1) pure (or glacial) acetic acid with stirring – this usually eliminates supercooling; and (2) pure acetic acid without stirring – supercooling will probably occur.

Procedure

Trial 1: With Stirring



1. Fasten a test tube clamp to a stand. Take a small-sized test tube and a stopper to the fume hood. Add 5 mL of acetic acid to the test tube and close the test tube with the stopper before removing from the fume hood.

2. Place the test tube in the test tube clamp to minimize spillage. Adjust the temperature of the acetic acid to about 25 ^{o}C by warming or cooling the test tube in a beaker of water.

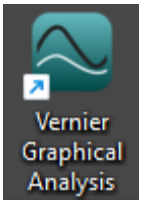
3. Prepare your cooling mixture by placing about 150 mL of ice in a 250 mL beaker; then add water to barely cover the ice.

4. Obtain the following equipment: Chemistry Department laptop (with Vernier Graphical Analysis app), Vernier LabQuest Mini, USB cable, thermometer probe.

5. Carefully insert the thermometer probe into the stopper & be sure the metal probe is touching the acetic acid.

6. Connect the thermometer probe to the LabQuest Mini (any CH) and then use the USB cable to connect the LabQuest Mini to the laptop.

7. Open the Vernier Graphical Analysis app. With the Temperature Probe connected, the window will show a graph of Temperature (°C). Time (s) and the current Temperature in the lower right of the window. Click on the button (should say "Mode Time Based") in the lower left corner for Data Collection Settings. Set the Time Units: min, Sample Rate: 10 samples / minute, End Collection: 10 min duration and click Done.



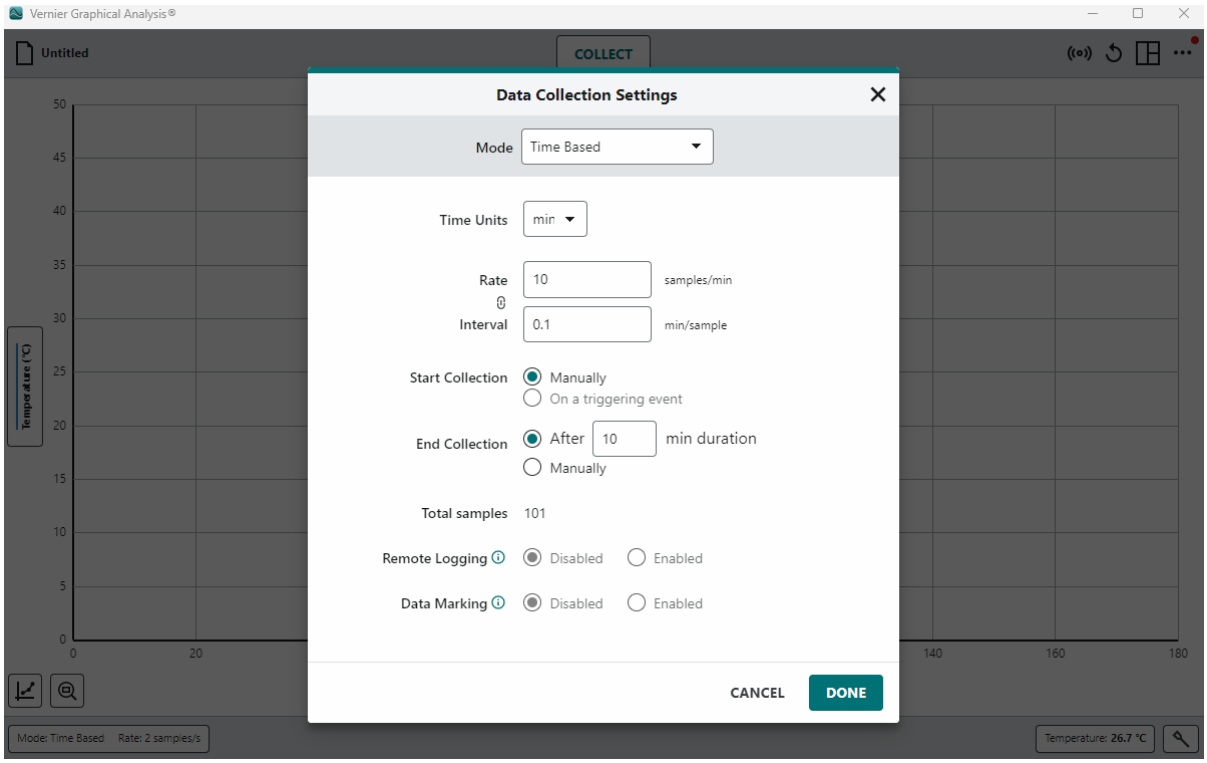


Figure 1. The Vernier Graphical Analysis Data Collection Settings for this experiment.

8. When you are ready to start the Trial, lower the test tube clamp until the acetic acid in the test tube is below the surface of the ice water and click the Collect button.

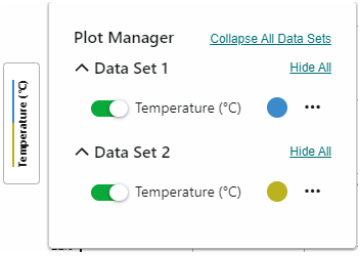
9. Use the thermometer as a gentle stirrer during this trial. Continue recording temperatures for 10 minutes where Vernier Graphical Analysis will stop automatically. If the temperature has stabilized for several minutes (flat part of the graph) and the acetic acid is completely frozen, you may click the Stop button to end the run.

10. To prepare the acetic acid for your next run, raise the test tube clamp and remove from the ice bath. Replace with a beaker of warm water to melt back to about 25 °C.

11. On the displayed graph, analyze the flat part. Press the mouse button and hold it down as you drag across the flat part to select it. Click Graph Options to View Statistics menu.

The mean temperature value for the selected data is listed in the statistics box on the graph. Record this value as the freezing temperature (in the Questions).

Trial 2: Without Stirring



1. For your next trial, Vernier Graphical Analysis will automatically store the run and start the next run when you click Collect in a new color of graph each time. To view the first trial with your second trail, click on the "Temperature (°C)" axis label and toggle on which Data Sets to view on the graph.

2. Replenish the ice bath if necessary.

3. Repeat the same timing procedure as Trial 1 but do not stir.

4. If the temperature falls to about 4 ^{o}C without forming crystals, you are observing supercooling. If supercooling persists for more than 2 minutes, "shock" the solution by moving the thermometer until crystals form.

5. Continue recording the temperature for the full 10 minutes or until completely frozen.

6. On the displayed graph, analyze the flat part using the Statistics function again and record this value as the freezing temperature in the Post Lab.

Completing the Graphs for your Experiment:

1. If not already both displayed, click the Temperature axis label, toggle on the 2 Data Sets to view on the same graph.
2. Label both curves by choosing Graph Options  Add Annotation, and "Stirring" (or "Without Stirring") in the edit box. Then drag each box to a position near its respective curve.

3. Be sure to show your graph of both Trial 1 and Trial 2 on the CHEM Dept. laptop to your instructor to receive credit for this part of the experiment OR they may instruct you to take a photo of your graph to submit with this report to Canvas.

<u>Hazardous Waste</u>

Thaw the acetic acid with a beaker of warm water and then remove the temperature probe. Using your wash bottle filled with water, rinse the temperature probe off into a beaker from your drawer. Take the stoppered test tube, your wash bottle, and the beaker to the fume hood. Pour the acetic acid out of the test tube into the provided "Recycled Acetic Acid" bottle. Before you leave the fume hood, use your wash bottle to rinse the acetic acid out of the test tube into your beaker. ***Please do not contaminate the recycled acetic acid with water!*** You can then pour the water that is in your beaker down the sink drain and wash your test tube and beaker before you put them away.

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Data and Calculations for Experiment 3

Use your graphs to answer the following questions. Note: The accepted freezing point of acetic acid, according to the *CRC Handbook of Chemistry and Physics*, is: 16.6 °C

1. a. What is the experimental freezing point of acetic acid in Trial 1? _____

   b. What is the experimental freezing point of acetic acid in Trial 2? _____

   c. Calculate the percent error in the Trial 1 measurement of the freezing point:

      SHOW CALCULATION:

      _____

2. What is supercooling? Did you observe supercooling in your experiment? Explain.

3. What is the difference between melting and freezing a substance?